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SPECIFICATION FOR BENZYL CHLORIDE, TECHN
(First Revision)

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1981

AMENDMENT NO. 1 MARCH 1983

TO

IS:1341-1980 SPECIFICATION FOR BENZYL CHLORIDE, TECHNICAL

(First Revision)

Corrigendum

[*Page 4, Table 1, Sl No.* (vi)] - Substitute the following for the existing matter under respective columns:

(1)	(2)	(3)	(4)
vi)	Benzylidine chloride ($C_6H_5CHCl_2$) content, percent by mass, *Max*	1.0	A-7

(PCDC 9)

Reprography Unit, ISI, New Delhi, India

IS : 5341 - 1980

Indian Standard
SPECIFICATION FOR BENZYL CHLORIDE, TECHNICAL
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(*Continued on page* 2)

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Indian Standard
SPECIFICATION FOR BENZYL CHLORIDE, TECHNICAL
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 10 August 1980, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1969. The Sectional Committee, responsible for the preparation of this standard reviewed it critically and decided to revise it in the light of the current testing techniques being followed in the industry. In this revision a chromatographic method for the determination of various impurities in the material has been introduced and the other methods of test examined and improved suitably. The sampling scheme has also been modified in this revision.

0.3 Benzyl chloride (α-Chlorotoluene) is a raw material for dyes, syntans, perfumery materials and pharmaceuticals. It is also used in the manufacture of photographic developer, synthetic resins, etc.

0.4 Benzyl chloride possesses acute local toxic hazard being lachrymatory and irritating to skin. It has a moderate fire and explosion hazard only when exposed to flame. All care, therefore, should be taken while handling this material.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for benzyl chloride, technical.

---

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be homogeneous, colourless or coloured liquid, very refractive, having rather unpleasant, irritating odour. It is insoluble in water; missible with alcohol, chloroform and ether. It rapidly decomposes when heated in presence of iron when it may result in violent explosion.

2.1.1 *Stabilizer* — To stabilize the material during transport, addition of soda ash is permissible, but it shall be not more than 0·1 percent by mass

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR BENZYL CHLORIDE, TECHNICAL

(*Clauses* 2.2 *and* B-5.1)

SL No (1)	CHARACTERISTIC (2)	REQUIREMENTS (3)	METHOD OF TEST (REF TO CL No. IN APPENDIX A) (4)
i)	Relative density* at 30°/30°C†	1·099 to 1·100	A-2
ii)	Distillation yield, percent by volume, *Min* at 175 to 185°C (the temperature being corrected for a pressure of 760 mmHg)	95	A-3
iii)	Evaporation residue, percent by mass, *Max*	0·050	A-4
iv)	Acidity of alkalinity	To pass the test	A-5
v)	Chlorine content, percent by mass,		A-6
	a) in the side chain	27·5 to 28·5	
	b) in the nucleus, *Max*	0·5	
vi)	Benzyl chloride ($C_6H_5CHCl_2$) content, percent by mass, *Max*	3·5	A-7
vii)	Assay (by GLC method), percent by mass, *Min*	98	A-7

*This term is used in the same sense as specific gravity with water as reference substance, as adopted by the International Organization for Standardization (ISO).

†The correction factor for relative density is +0·000 74 for each degree celsius fall, and −0·000 74 for each degree celsius rise in temperature within the temperature range of 20 to 40°C.

3. PRECAUTIONS IN HANDLING

3.1 The drums or carboys shall be stored in a cool, well ventilated store, where no naked flame shall be allowed. The material being lachrymatory all care shall be taken to prevent contact with vapours (*see also* **0.4**).

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979*) or glass carboys duly provided in hampers, as agreed to between the purchaser and the supplier.

4.2 Marking — The material shall be marked with the following information apart from the minimum cautionary note worded as 'WARNING : HAZARDOUS LIQUID — LACHRYMATORY' :

a) Name of the material,
b) Manufacturer's name and his recognized trade-mark, if any;
c) Net, gross and tare mass of the material; and
d) Lot or batch number, in code or otherwise.

4.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to the requirements of this standard be judged as prescribed in Appendix B.

---

*Specification for steel drums (galvanized and ungalvanized) (*second revision*)

APPENDIX A

(*Clause* 2.2 and *Table* 1)

METHODS OF TEST FOR BENZYL CHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977[*]) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 Outline of the Method — In this method, mass of equal volumes of the material and water are compared.

A-2.1 Apparatus

A-2.1.1 *Relative Density Bottle* — about 25 ml capacity.

A-2.1.2 *Water Bath* — maintained at 30·0 ± 0·2°C.

A-2.1.3 *Thermometer* — any convenient thermometer of a suitable range with 0·1 degree or 0·2 degree graduation (*see* IS : 2480-1973[†]).

A-2.2 Procedure — Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 30°C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the water bath, keep the entire bulb completely covered with water and maintain the bath temperature at 30·0 ± 0·2°C for 30 minutes. Wipe carefully any water exuded from the capillary opening. Remove from the bath, wipe completely dry and weigh. Calculate the mass of water. Again cool and carefully dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-2.3 Calculation

$$\text{Relative density at } 30°/30°\text{C} = \frac{A - B}{C - B}$$

---

[*] Specification for water for general laboratory use (*second revision*).

[†] Specification for solid stem general purposes glass thermometers (*first revision*).

where

A = mass in grams of the relative density bottle with the material,

B = mass in grams of the relative density bottle, and

C = mass in grams of the relative density bottle with water.

A-3. DETERMINATION OF DISTILLATION YIELD

A-3.1 Apparatus

A-3.1.1 *Distillation Flask* — of the shape and dimensions given in Fig 1 Fix the flask in the vertical position by means of a clamp at the extreme upper end of the neck

A-3.1.2 *Thermometer* — any suitable thermometer conforming to the following requirements.

Range	95 to 255°C
Graduation	0·5°
Immersion	100 mm
Overall length, *Max*	385 mm
Stem diameter	5·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	15 to 20 mm
Bulb diameter	Not greater than stem
Distance from bottom of bulb to the bottom of scale, *Min*	125 mm
Scale error, *Max*	1·0°

A-3.1.2.1 The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

A-3.1.3 *Liebig Condenser* — made of type 1 glass (graded according to IS : 2303-1963*) with a wall thickness of 1·0 to 1·5 mm and conforming to the shape and dimensions given in Fig 2.

A-3.1.3.1 Alternatively, the bent portion may be substituted by an adapter fitted externally such that the distillate does not come in contact with the cork.

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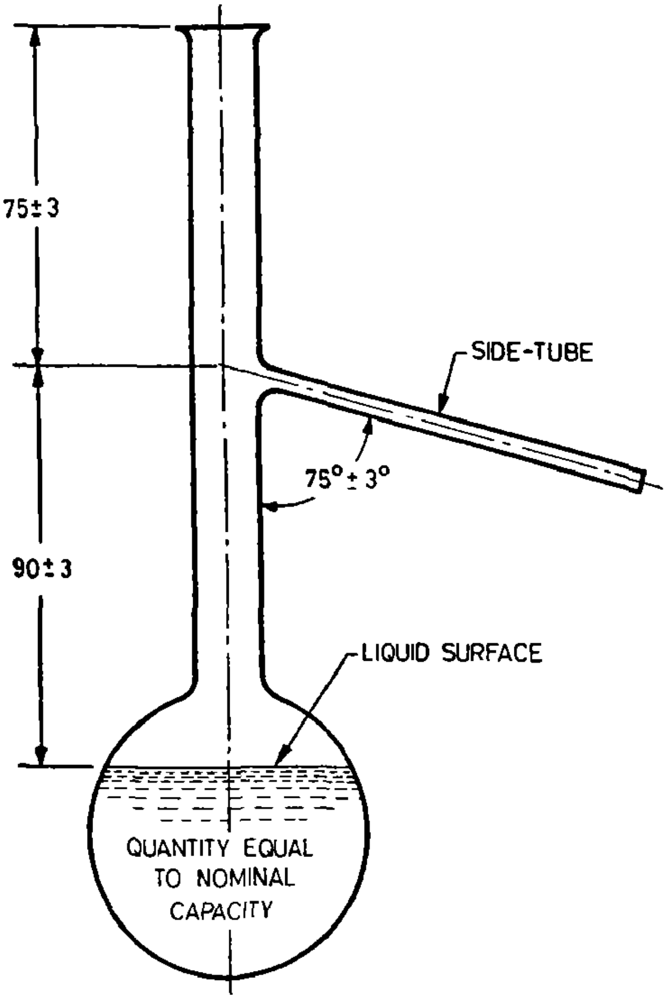
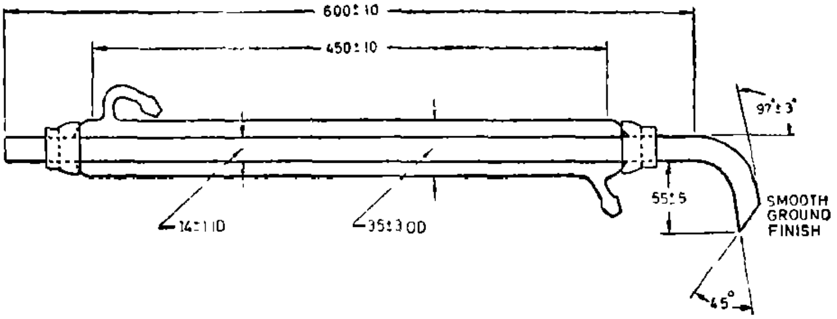
*Methods of grading glass for alkalinity.



All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK



All dimensions in millimetres.

FIG. 2 LIEBIG CONDENSER

A-3.1.4 *Receiver* — 100-ml capacity with 1-ml marks running half way round the circumference, 5-ml marks running three-quarters way round, and 10-ml marks running all round the circumference and numbered (*see* Fig 3). A graduated cylinder of 100 ml capacity may also be used

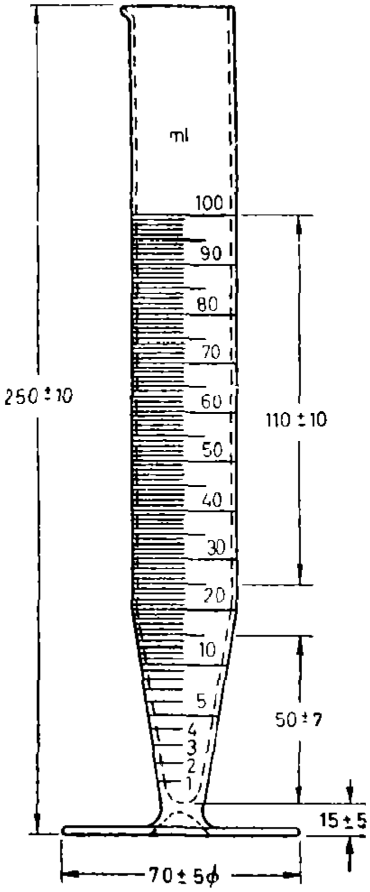
A-3.1.5 *Rectangular Draught Screen* — made of 0 8 mm sheet metal, with the dimensions shown in Fig 4, open at the top and bottom and fitted with hard asbestos board with a central hole of 110 mm diameter It shall comply with the following requirements

a) In each of the two narrower sides of the draught screen there shall be two circular air-vent holes, each 25 mm in diameter, situated below the asbestos shelf as shown in Fig. 4. In each of the four sides of the draught screen there shall be 3 air-vent holes with their centres 25 mm above the base of the draught screen These holes shall occupy the position as shown in Fig 4 The diameter of each of the holes centrally situated in the longer sides shall be 25 mm and of the remaining ten holes 12 5 mm. At the middle of each of the wider sides, a vertical slot with the dimensions shown in Fig. 4 shall be cut downwards from the top of the screen. A removable shutter conforming to the dimensions in Fig 5 shall be provided for closing whichever vertical slot is not in use.

b) A shelf of hard asbestos, 6 mm in thickness and having a central circular hole 110 mm in diameter, is supported horizontally in the screen and fits closely to the sides of the screen, to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

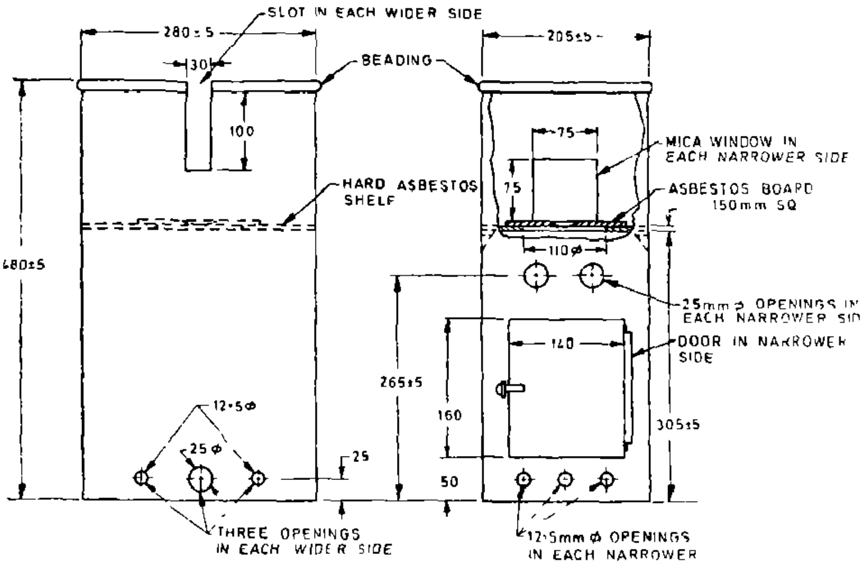
c) In one of the narrower sides of the screen, a door shall be provided having the position and dimensions shown in Fig. 4 and overlapping an opening in the screen by approximately 5 mm all round In each of the narrower sides of the screen, a mica window shall be placed centrally with the bottom of the window level with the top of the asbestos shelf. The dimensions and position of windows are shown in Fig. 4.

d) *Asbestos Board* — In addition to the asbestos shelf referred to in the previous section, an asbestos board 150 mm square, 6 mm in thickness, is required. It shall have a central hole of 50 mm diameter



All dimensions in millimetres.

FIG. 3 RECEIVER



All dimensions in millimetres.

FIG. 4 DRAUGHT SCREEN

A-3.1.6 *Bunsen Burner, Electric Heater or Other Flame Type of Heater* — Any type of heater or burner may be employed that enables the distillation to be carried out as prescribed in A-3.3

A-3.2 Assembly of Apparatus — Assemble the apparatus as shown in Fig. 6

A-3.2.1 *Position of Thermometer* — The thermometer shall be held concentrically in the neck of the flask by means of a well-fitting stopper of a material which is not attacked by the liquid, and the junction of the capillary tube and the main bulb of the thermometer is maintained level with the lower edge of the joint between the side tube and the neck of the flask. When the thermometer is fixed in position as indicated above, the immersion line on the thermometer shall be in the neighbourhood of the top of the cork holding the thermometer in position in the neck of the flask.

A-3.2.2 *Support for Flask* — The asbestos board [*see* A-3.1.5(d)] shall be on the top of the asbestos shelf of the draught screen so that the two holes are approximately concentric. The flask shall then be placed in position and pressed down so as to close completely the hole in the asbestos board.

A-3.2.3 *Connection of Flask to Condenser* — The flask is connected to the condenser so that the end of the side tube projects at least 25 mm into the condenser and is coaxial with it.

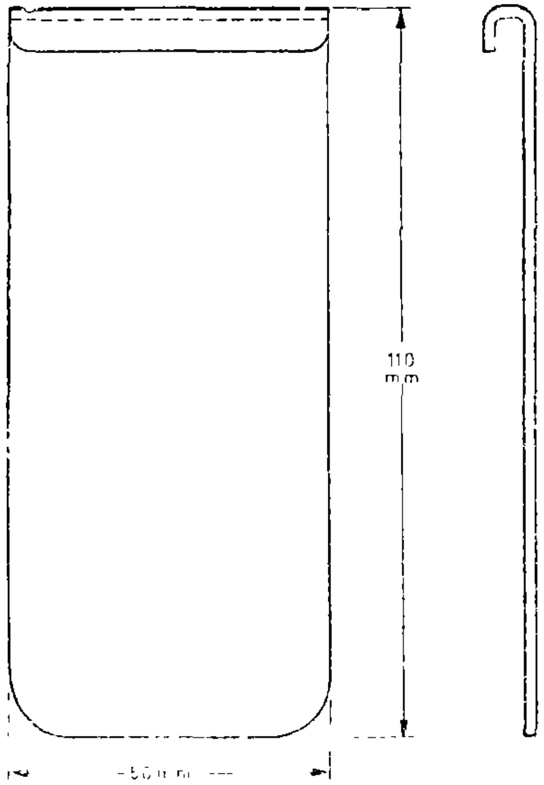


FIG. 5 REMOVABLE SHUTTER

A-3.3 Procedure

A-3.3.1 Measure 100 ml of the material at laboratory temperature into the graduated cylinder and transfer it to the distillation flask, the contents of the graduated cylinder being allowed to drain for 15 seconds into the flask. Add a fragment of a porous material (about 2 mm cube) or some other suitable inert material to avoid bumping, connect the flask to the condenser and insert the thermometer Pass an adequate supply of cold water through the condenser. To receive the distillate, use the 100-ml receiver in which the sample was measured, without rinsing or drying Heat the flask slowly especially after the ebullition has begun in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate drops into the receiver, care being taken that the total period of this preliminary heating is neither less than 12 nor longer than 17 minutes. Place the receiver so that the condensate flows down its inside walls. Continue the distillation at the rate of 4 to 5 ml per minute by suitable regulation of flame and take the readings of volume at the specified temperature.

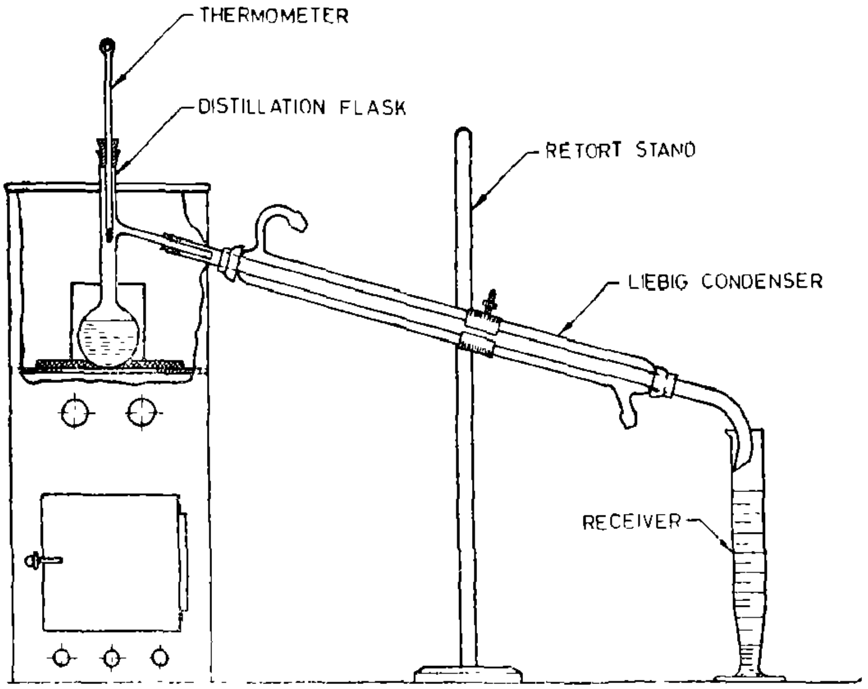


FIG. 6 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF DISTILLATION RANGE

A-3.3.2 Read the volume of distillate in the receiver when the thermometer indicates each of the correct specified distillation temperatures The temperature on the thermometer scale shall be corrected as specified under **A-3.4**, before commencing the distilltaion. The difference between the volumes so recorded is the volume distilling between the specified temperatures at 760 mmHg pressure

A-3.4 Correction of Thermometer Reading

A-3.4.1 *Error of Scale* — In all thermometers make the corrections as indicated on the certificate of the instrument.

A-3.4.2 *Correction for Barometric Pressure* — If the barometric pressure prevailing during the determination is 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected under **A-3.4.1** shall be used as such. If the prevailing barometric pressure deviates from 760 mmHg, the specified temperature shall be corrected with the correction factor given below:

a) For every 10 mm above 760 mmHg, subtract 0·5° from the specified temperature, and

b) For every 10 mm below 760 mmHg, add 0·5° to the specified temperature

NOTE — These corrections are valid only for pressures above 700 mmHg.

A-4. DETERMINATION OF RESIDUE ON EVAPORATION

A-4.1 Apparatus

A-4.1.1 *Basin* — porcelain or silica.

A-4.1.2 *Water Bath*

A-4.1.3 *Oven* — capable of maintaining temperature of 180 ± 5°C.

A-4.2 Procedure — Evaporate 50 ml of the material, accurately weighed, in a tared porcelain or silica basin over a water bath to dryness. Wipe the bottom and dry the residue in an oven at 180 ± 5°C for 2 hours Cool in a desiccator and weigh.

A-4.3 Calculation

$$\text{Residue on evaporation, percent by mass} = 100 \times \frac{A - B}{C - B}$$

where

A = mass in g of the basin with the residue,

B = mass in g of the basin, and

C = mass in g of the basin with the material to be tested.

A-5. TEST FOR ACIDITY OR ALKALINITY

A-5.1 Reagents

A-5.1.1 *Sodium Chloride Solution* — saturated, neutral to methyl orange.

A-5.1.2 *Methyl Orange Indicator* — Dissolve 0 1 g of methyl orange in 100 ml of water.

A-5.1.3 *Standard Sodium Hydroxide Solution* — 0 1 N.

A-5.2 Procedure — Take 20 ml of sodium chloride solution in a 250-ml conical flask, add 5 ml of the material and two drops of methyl orange After stirring add one drop (0 05 ml) of standard sodium hydroxide solution.

A-5.2.1 The material shall be regarded to have passed the test if the solution does not have red colour after the addition of sodium hydroxide solution.

A-6. DETERMINATION OF CHLORINE CONTENT

A-6.0 Outline of the Method — The material dissolved in *iso*-propyl alcohol is treated with metallic sodium and refluxed. Under the conditions the organically bound chlorine gets converted to ionisable sodium chloride. The contents are taken up with water, acidified and treated with known volume of silver nitrate solution. The total chlorine is then determined from the amount of silver nitrate solution used up by titrating the excess silver nitrate solution against standard ammonium thiocyanate solution using ferric alum solution as indicator. Similarly, the chlorine in side chain is determined by refluxing the material with ethanolic sodium hydroxide solution and following the above procedure. The chlorine in the nucleus is determined by subtracting the chlorine in side chain from total chlorine.

A-6.1 Reagents

A-6.1.1 *Iso-propyl Alcohol* — conforming to IS : 2631-1976*.

A-6.1.2 *Aqueous Iso-propyl Alcohol* — 50 percent (*v/v*).

A-6.1.3 *Metallic Sodium* — pure, in the form of ribbon or cut in small pieces.

A-6.1.4 *Dilute Nitric Acid* — 1 : 3 (*v/v*).

A-6.1.5 *Standard Silver Nitrate Solution* — 0.1 N (*see* IS : 2316-1968†).

A-6.1.6 *Ferric Alum Indicator Solution* — saturate or dissolve 8 g of ferric ammonium sulphate in 400 ml of water containing 3 to 5 ml of concentrated sulphuric acid (*see* IS : 266-1977‡).

A-6.1.7 *Nitrobenzene*

A-6.1.8 *Standard Ammonium Thiocyanate Solution* — 0.1 N (*see* IS : 2316-1968†).

A-6.1.9 *Standard Ethanolic Sodium Hydroxide Solution* — approximately 0.5 N. Free from chlorine.

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*Specification for *iso*-propyl alcohol (*first revision*).

†Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

‡Specification for sulphuric acid (*second revision*).

A-6.2 Procedure

A-6.2.1 *Total Chlorine* — Take 50 ml of *iso*-propyl alcohol in a 250-ml flask and add 0·2 to 0·3 g of material accurately weighed. Add 3 g of metallic sodium slowly, till there is no reaction. Connect the flask to a reflux condenser and boil vigorously for 1 hour, shaking the flask occasionally. Dissolve the excess metallic sodium by cautiously adding 10 ml of aqueous *iso*-propyl alcohol through the condenser at the rate of one or two drops per second. Boil for additional 10 minutes and then add about 50 ml of water. Cool to room temperature and disconnect the flask from condenser. Acidify the contents with dilute nitric acid until just acidic (blue litmus paper turning red). Add exactly 50 ml of silver nitrate solution, followed by 50 ml of nitrobenzene (*see* Note 1) and 2 to 3 ml of ferric alum indicator solution.

Add standard ammonium thiocyanate solution slowly from the burette to the contents in the flask, constantly swirling. At the first appearance of red colour, stopper the flask tightly and shake vigorously for 15 seconds to coagulate and remove any precipitate from aqueous phase (*see* Note 2).

Continue the titration slowly until the end point of brick red colour of aqueous phase is reached. Carry out a blank determination simultaneously following exactly the procedure used for the material.

NOTE 1 — Nitrobenzene is very hazardous when absorbed through skin or when its vapour is inhaled. Such exposure may cause anemia. Do not get nitrobenzene in the eyes, on the skin or on clothing. Avoid breathing its vapour. Use only with adequate ventilation.

NOTE 2 — Care should be exercised in opening the flask after shaking. Pressure may develop causing small amounts of acid solution to be sprayed from the mouth of the flask as the stopper is removed.

A-6.2.1.1 *Calculation*

$$\text{Total chlorine (as Cl), percent by mass } (X_1) = \frac{(V_1 - V_2)\, N \times 3{\cdot}546}{M}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution used in the blank determination,

V_2 = volume in ml of standard ammonium thiocyanate solution used in the test with the material,

N = normality of standard ammonium thiocyanate solution, and

M = mass in g of the material taken for the test.

A-6.2.2 *Chlorine in Side Chain* — Weigh accurately 0·2 to 0·3 g of the material and take in a 250-ml flask, provided with a ground neck. Add 50 ml of standard ethanolic sodium hydroxide solution and reflux for 2 hours.

Cool the solution, acidify with dilute nitric acid. Add 50 ml of silver nitrate solution followed by 10 ml of nitrobenzene (*see* Note 1 under **A-6.2.1**), and 2 to 3 ml of ferric alum solution indicator Titrate the excess silver nitrate solution against standard ammonium thiocyanate solution to the end point of appearance of brick red colour of the aqueous phase Carry out a blank determination.

A-6.2.2.1 *Calculation*

$$\text{Chlorine in side chain, percent by mass } (X_2) = \frac{(V_1 - V_2) N \times 3{\cdot}546}{M}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution used in the blank determination,

V_2 = volume in ml of standard ammonium thiocyanate solution used in the test with the material,

N = normality of standard ammonium thiocyanate solution, and

M = mass in g of the material taken for the test.

A-6.2.3 Chlorine in the nucleus, percent by mass = $X_1 - X_2$

where

X_1 = total chlorine, and

X_2 = chlorine in the side chain.

A-7. METHOD FOR DETERMINATION OF IMPURITIES IN PURE BENZYL CHLORIDE

A-7.0 General — This method is intended for the quantitative determination of toluene, chlorotoluenes and benzylidine chloride in benzyl chloride The method is intended to cover the range of 0·02 to 1·00 percent (*m/m*) of each type of impurities

A-7.1 Outline of Method — A known amount of internal standard is added to the sample and an aliquot portion of this mixture is introduced into the separating column of a gas chromatograph by means of a syringe. The vaporized sample is swept through a column by a flow of carrier gas and as each component emerges, it is detected by a flame ionization detector and recorded as a peak in a chromatogram. Under the operating conditions the impurities are identified by their relative retentions and are quantitatively determined from their peak areas relative to the peak area of the internal standard.

A-7.2 Reagents

A-7.2.1 *Toluene*

A-7.2.2 *θ-Chlorotoluene*

A-7.2.3 *Benzylidine Chloride*

A-7.2.4 *2-Ethyl Hexanol* — as solvent in calibration blend.

A-7.2.5 *Acetic Acid* — as internal standard.

NOTE — The purity of the chemicals used should be more than 99 percent

A-7.3 Apparatus

A-7.3.1 *Gas Chromatograph* — Any gas chromatograph fitted with a flame ionization detector and capable of meeting the required operating conditions may be used. The instrument shall have sufficient sensitivity to obtain a peak height of at least twice the noise level for 0.005 percent *m/m* toluene under the specified operating conditions

NOTE — It is recommended that maximum resolution should be obtained in the injection column If an inlet splitter is fitted to the chromatograph, poor resolution may result from blocking it at the vent

A-7.3.2 *Integrator* — Electronic integrator is recommended but other methods of recording and measuring peak areas may be used, provided that the sensitivity requirements are met.

A-7.3.3 *Recorder* — A strip chart recorder with a scale of at least 250 mm and a response time of not more than one second for full scale deflection.

A-7.3.4 *Packed Columns* — As per the manufacturer's specifications.

A-7.4 Procedure — The operating conditions in the column shall be as follows:

a) Column of stainless steel or glass, 1 m long and 0.6 mm outer diameter approximately with 5 percent carbowax, 20 percent terephthalic acid on AW-DMCS chromosorb G.

b) Column temperature	95°C
c) Injection temperature	235°C
d) Carrier gas	Nitrogen
e) Carrier flow rate	Adjust to give a retention time for benzyl chloride between 2 min and 4 min or 40 ml/min
f) Fuel gases	Hydrogen and air flow rates for the flame ionization detector shall be adjusted to the levels recommended by the instrument manufacturers

g) Internal standard	Acetic acid
h) Sample size	0·2 μl is recommended but larger samples may be used provided that satisfactory resolution is maintained
j) Chart speed	Not less than 40 mm/min if the peak areas are measured manually

A-7.4.1 *Column Performance* — The column shall be capable of achieving the following performance when operated under conditions specified in the method:

a) The internal standard shall be completely resolved from all other peaks in the chromatogram

> NOTE — If necessary, resolution of the internal standard from other components may be achieved by adjusting the column temperature up to 20°C above or below the recommended temperature of 95°C

b) Test the performance of the column by preparing a blend of 0·1 percent (*v/v*) of toluene in 2-ethyl hexanol. Inject a sample of the blend of same size as used in the analytical methods into the chromatograph and record the chromatogram The height of the valley between the two peaks measured from the base line shall be not more than 2 percent of the toluene peak if integration is used, and not more than 10 percent if measurement is done by hand

A-7.4.2 *Calibration*

a) Prepare a calibration blend of acetic acid, toluene, *o*-chlorotoluene and benzylidine chloride in 2-ethyl hexanol as follows:

> Add 11·0 ml of 2-ethyl hexanol, 0·05 ml (about 1 drop by 1 ml pipette) each of toluene and *o*-chlorotoluene, 0·1 ml benzylidine chloride and 0·5 ml acetic acid, in that order into a clean dry stoppered flask, weighing the flask before and after each addition. Shake well and use for injection Weighings shall be made up to ± 0·2 mg accuracy The calibration blend shall be prepared just before use

b) Prepare the sample as follows.

> Add 0·5 ml acetic acid and 8·7 ml sample into another clean, dry stoppered flask, weighing the flask before and after each addition Shake well and use for injection Inject the calibration blend and the prepared sample into the chromatograph under the specified conditions (*see* **A-7.3.4**)

The components are identified from the typical chromatogram as shown in Fig 7 and from the relative retention times given under **A-7.5**.

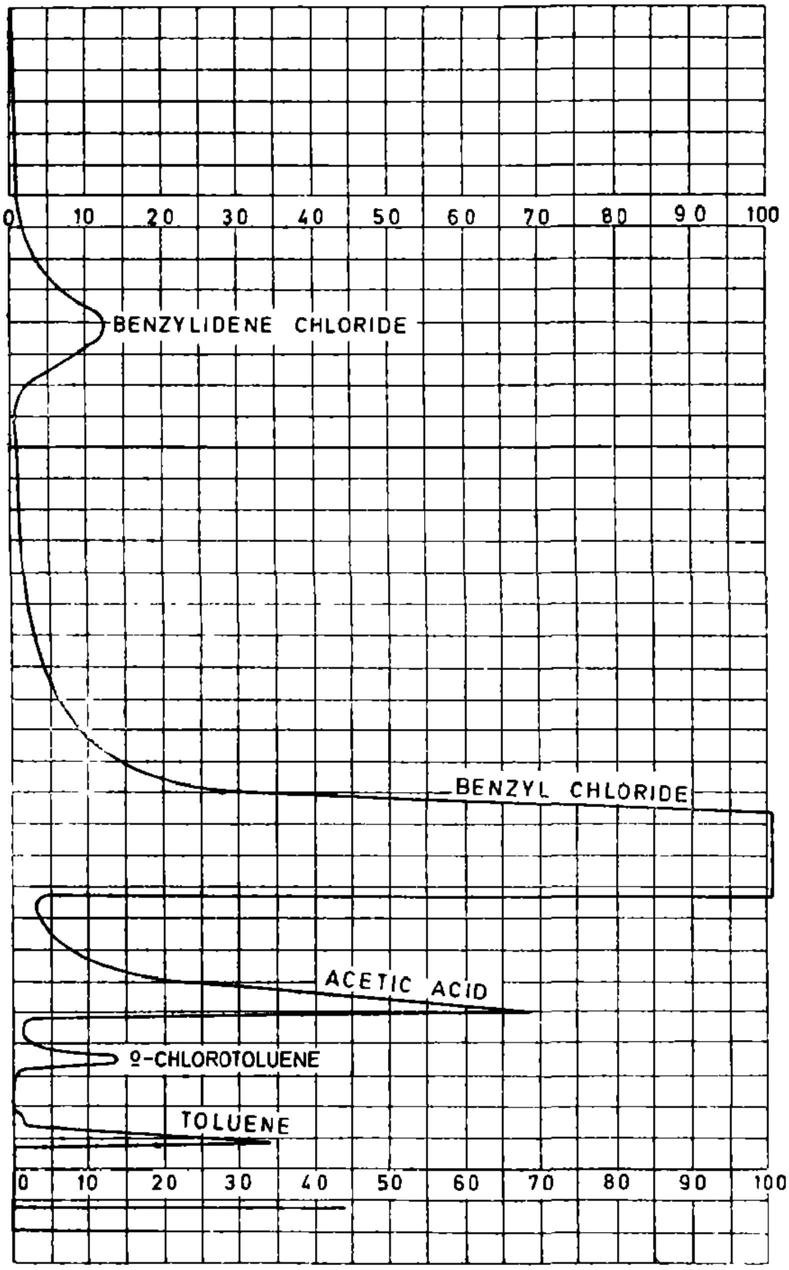


FIG. 7 TYPICAL CHROMATOGRAM OF BENZYL CHLORIDE

A-7.5 Typical relative retention times of the components are as follows:

Toluene	0·33
o-chlorotoluene	0·74
2-Ethyl hexanol	1·40
Benzyl chloride	1·70
Benzylidine chloride	4·53

A-7.6 Calculation — Concentration of impurities expressed as percentages by mass are given by the formula

$$\frac{100 \times A_i \times a_c \times m_i \times M_c}{A_c \times a_i \times m_t \times M_i}$$

where

A_i = peak area of internal standard in calibration blend,
a_c = peak area of component in sample,
A_c = peak area of component in calibration blend,
a_i = peak area of internal standard in sample,
m_i = mass in g of internal standard in sample,
M_c = mass in g of component in calibration blend,
m_t = total mass in g of sample, and
M_i = mass in g of internal standard in calibration blend

NOTE — Report the results for toluene, *o*-chlorotoluene and benzylidine chloride to the nearest 0·01 (*m/m*)

A-7.6.1 Assay (as benzyl chloride), percent by mass = 100 — (sum of the impurities).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF BENZYL CHLORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination (*see* **3.1**).

B-1.4 To draw a representative sample, the contents of each container selected for sampling, shall be mixed as thoroughly as possible, by suitable means.

B-1.5 The samples shall be placed in a clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.0 The following forms of sampling instruments may be used

a) Sampling bottle or can for taking samples from various depths in large tanks, and
b) Sampling tube.

B-2.1 Sampling Bottle or Can — It consists of a weighted bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig 8) The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

B-2.2 Sampling Tube — It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig 9) The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached It is then opened for a short time to admit the material and finally closed and withdrawn.

B-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2

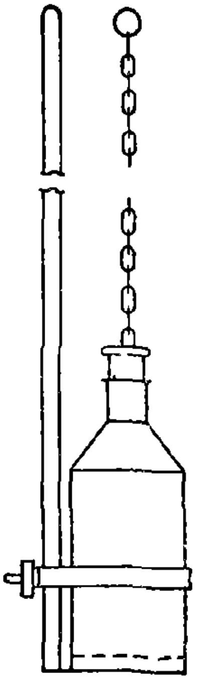
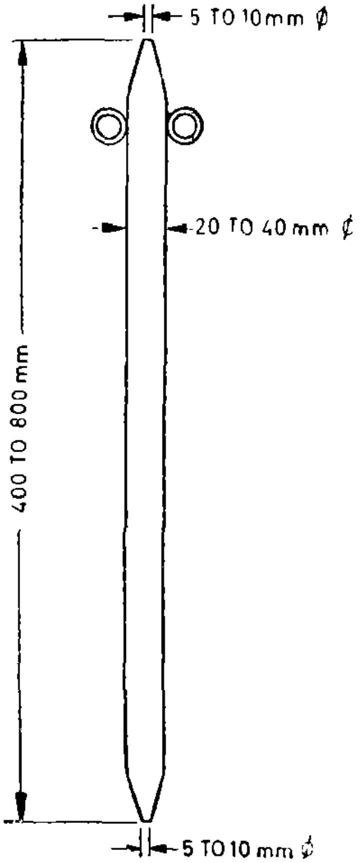


FIG 8 SAMPLING BOTTLE OR CAN



All dimensions in millimetres

FIG. 9 SAMPLING TUBE

B-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. In case no such table is available, the following procedure is recommended for use:

> Starting from any container from the lot, count them as 1,2,3,......, etc. up to r and so on in one order where r is the integral part of N/n (N being the number of containers in the lot and n being the number of containers to be selected). Every rth container thus counted shall be withdrawn to give samples for tests.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-3.2)

LOT SIZE N	NO. OF CONTAINERS TO BE SELECTED n
Up to 100	5
101 ,, 200	6
201 ,, 300	7
301 ,, 400	8
401 ,, 500	9
501 and above	10

NOTE — In the case of very small lots where the selection of five containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of this specification shall be as agreed to between the purchaser and the supplier

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected as in **B-3.2**, a representative portion of the material from different parts of the container shall be drawn with the help of a suitable sampling instrument (*see* **B-2**).

B-4.2 From each of these individual portions (*see* **B-4.1**) an equal quantity of material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 000 ml in volume. The composite sample shall be divided into three equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample. These shall be transferred to separate containers and shall be sealed and marked with full identification particulars given under **B-1.7**.

B-5. TESTS

B-5.1 Tests for the determination of all the characteristics given in **2.1** and Table 1 shall be conducted on the composite sample.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For declaring the conformity of a lot to the requirements of all the characteristics, the test result for each of the characteristics shall satisfy the corresponding requirement specified in the standard.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	$1 \text{ N} = 1 \text{ kg. m/s}^2$
Energy	joule	J	$1 \text{ J} = 1 \text{ N.m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V.s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s } (\text{s}^{-1})$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volt	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$

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